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METHOD 525.2

I. SCOPE AND APPLICATION:

This is a gas chromatographic (GC) method applicable to the determination of certain chlorinated acids in ground water and finished drinking water. The following compounds can be determined using this method:

Analyte	Chemical Abstract Services Registry Numbers (CASRN)
Acenaphthylene	208-96-8
Alachlor	15972-60-8
Aldrin	309-00-2
Anthracene	120-12-7
Atrazine	1912-24-9
Benz[a]anthracene	56-55-9
Benzo[b]fluoranthene	205-82-3
Benzo[k]fluoranthene	207-08-9
Benzo[a]pyrene	50-32-8
Benzo[g,h,i]perylene	191-24-2
Butylbenzylphthalate	85-68-7
Chlordane components	
Alpha-chlordane	5103-71-9
Gamma-chlordane	5103-74-2
Trans nonachlor	39765-80-5
2-Chlorobiphenyl	2051-60-7
Chrysene	218-01-9
Dibenz[a,h]anthracene	53-70-3
Di-n-butylphthalate	84-72-3
2,3-Dichlorobiphenyl	16605-91-7
Diethylphthalate	84-66-2
Bis(2-ethylhexyl) adipate	103-23-1

Analyte	Chemical Abstract Services Registry Numbers (CASRN)
Bis(2-ethylhexyl) phthalate	117-81-7
Dimethylphthalate	131-11-3
Endrin	72-20-8
Fluorene	86-73-7
Heptachlor	76-44-8
Heptachlor epoxide	1024-57-3
2,2',3,3',4,4',6-Heptachlorobiphenyl	52663-71-5
Hexachlorobenzene	118-74-1
2,2',4,4',5,6'-Hexachlorobiphenyl	60145-22-4
Hexachlorocyclopentadiene	77-47-4
Indeno[1,2,3,c,d]pyrene	193-39-5
Lindane	58-89-9
Methoxychlor	72-43-5
2,2',3,3',4,5',6,6'-Octachlorobiphenyl	40186-71-8
2,2',3',4,6-Pentachlorobiphenyl	60233-25-2
Pentachlorophenol	87-86-5
Phenanthrene	85-01-8
Pyrene	129-00-0
Simazine	122-34-9
2,2',4,4'-Tetrachlorobiphenyl	2437-79-8
Toxaphene mixture	8001-35-2
2,4,5-Trichlorobiphenyl	15862-07-4

II. REAGENTS:

- Sodium Thiosulfate ($Na_2S_2O_3$) solution
- 1:1 hydrochloric acid (HCL) solution

III. MATERIALS:

- 1-liter amber borosilicate sample bottle fitted with screw caps lined with TFE-fluorocarbon.
- Latex gloves
- Protective eyewear
- Plastic container for disposal of used pipette tips
- Disposable glass pipette and rubber bulb.
- Pool and Spa 3-Way Test Strips (Chem Lab Products, Inc.)
- Kim wipes and Paper Towels
- Pliers

IV. PROCEDURE:

1. Remove any attachments such as hoses, screens or aeration devices on the faucet. Inspect the faucet for anything that may fall into the sample container.

2. Open the tap and allow the system to flush for about 10 minutes. This should be sufficiently long enough to allow the water temperature to stabilize and get a representative sample.

3. Adjust the water flow to about 1000 ml/minute or slow enough that no air bubbles purge the sample when collecting from the flowing stream.

4. Remove the cap from the 1-liter container. Do not rinse the container as it has already been acid rinsed and may already contain sodium thiosulfate as a preservative.

5. To fill, tip the bottle to about a 45° angle into the stream of water. Ensure the stream is sufficiently slow so as to be able to anticipate when the bottle is nearly full and thus avoid over flowing. Fill the bottle to within approximately ½ inch of the mouth.

6. Remove the bottle from the flow and recap. Invert the sample bottle five times.

7. Place a chlorine detector strip on a dry opened paper towel. Remove the screw-on cap and obtain an aliquot of the sample using a glass pipette. Moisten the chlorine detector strip with the aliquot from the glass pipette and immediately flick the chlorine detector strip once using a sharp wrist motion to shake off the excess water. Compare the strip with the reference chlorine range. A determination must be made within 30 seconds.

8. If no chlorine is detected, record results in field notebook and advance to acidification step using 1:1 HCl in item #13.

9. If chlorine is present, add 5 drops of sodium thiosulfate solution, recap the bottle firmly and invert 5 times. Place a chlorine detector strip on a dry opened paper towel.

10. Remove the screw-on cap and obtain an aliquot of the sample using a glass pipette. Thoroughly moisten the chlorine detector strip with the aliquot from the glass pipette and immediately flick the chlorine detector strip once using a sharp wrist motion to shake off the excess water. Compare the strip with the reference chlorine range. A determination must be made within 30 seconds.

11. If no chlorine is detected, record the results in the field notebook and proceed to the acidification step with 1:1 HCl in item #13.

IV. PROCEDURE (continued):

12. Continue the process of adding sodium thiosulfate to the sample, recapping, mixing, and testing until no chlorine is detected. Remember to note the number of drops of sodium thiosulfate added to the water sample in the field notebook.

13. The sample must now be tested for pH concentration. Begin by adding 10 drops of 1:1 HCl (0.5 ml) to the sample and capping. Invert three times and uncap. Dip a strip of pH test paper indicator into the experimental sample and remove, giving the test strip a quick flick of the wrist to shake off excess water. Compare the color change to the reference chart. Determining the pH must be accomplished within a 30 second period. The sample must be acidified to a pH of ≤ 2. If the sample is adequately preserved, recap the bottle firmly and record the results in the field notebook. Dry the sample bottle, attach the sample/laboratory label to the bottle and secure the chain of custody seal around the cap. Place the sample bottle in the ice chest to cool to 4°C. Duplicate samples not required.

14. If the pH is higher than 2, add 5 drops of 1:1 HCl using a clean glass pipette to the sample, recap, and invert three times.

15. Uncap the vial and retest using a fresh pH test strip.

16. If the pH is ≤ 2, then record in the field notebook the number of drops need to adequately acidify the sample and place in the ice chest for transportation. If the pH is > 2, continue the cyclic procedure of adding 5 drops of HCl, capping, inverting three times, uncapping and retesting using a fresh pH strip until the sample is adequately preserved. Determining the pH must be accomplished within a 30 second period. Record the final number of drops required by the sample to acidify to a pH ≤ 2 in the field notebook. Dry the sample bottle, attach the sample label to the bottle and secure the chain of custody seal around the cap. Place the sample bottle in the ice chest to cool to 4°C. Duplicate samples are not required.

V. SAMPLE TRANSPORT:

After obtaining the water samples, attach the completed chain of custody seal around the plastic cap of each 1-liter bottle. The 1-liter bottle must be amber colored to reflect sunlight since some of the pesticides analyzed for in this method are light sensitive and degrade when exposed to ultraviolet radiation. Place the sample bottle into the ice chest for transport. The samples must be chilled and preserved at a temperature of 4°C and maintained at that temperature until analysis. Always use chopped, grated, or dry ice when chilling the voa samples for transportation. Never use "blue ice" as the samples will not adequately chill. Field samples that will not be received at the laboratory on the day of collection must be packaged for shipment with sufficient ice to ensure they will be at 4°C upon arrival at the laboratory.

VI. SAMPLE PRESERVATION:

The samples must be iced or refrigerated at 4°C and protected from light from the time of collection until extraction. Preservation study results indicate that the sample analytes (measures as total acid), except for 5-hydroxydicamba, are stable in water for 14 days when stored under these conditions. It has been demonstrated that the concentration of 5-hydroxydicamba substantially degrades over a span of 14 days in a biologically active matrix.

VII. DEFINITIONS:

A. *Sodium Thiosulfate ($Na_2S_2O_3$):* A preservative use to dechlorinate water samples. Reduces free chlorine into acid.

B. *Eluant:* The solvent that contains the analytes after extraction or desorption.

VIII. SAFETY:

The use of protective eyewear and laboratory quality latex gloves is highly recommended when collecting and preserving samples.

IX. SUMMARY OF METHOD:

METHOD 525.2: Organic compound analytes, internal standards, and surrogates are extracted from a water sample by passing 1 liter of sample water through a cartridge or disk containing an inert solid matrix with a chemically bonded C_{18} organic phase (liquid/solid extraction, LSE). After the target compounds are adsorbed to the C_{18} organic phase, these organic compounds are desorbed (eluted) from the LSE cartridge or disc with small quantities of ethyl acetate followed by methylene chloride, and this extract is concentrated further by evaporation of some of the solvent.

The sample components are separated, identified, and measured by injecting a 2µl aliquot of the concentrated extract into a high resolution fused silica capillary column of a gas chromatography/mass spectrometry (GC/MS) system. Compounds eluting from the GC column are identified by comparing their measured mass spectra and retention times to the reference spectra and retention times in the database library.

Reference spectra and retention times for analytes are obtained by the measurement of calibration standards under the same conditions used for sample analysis. The concentration of each identified component is measured by relating the MS response of the quantitation ion produced by that compound to the MS response of the quantitation ion produced by a compound that is used as an internal standard. Surrogate analytes, whose concentrations are known in every sample, are measured with the same internal standard calibration procedure.